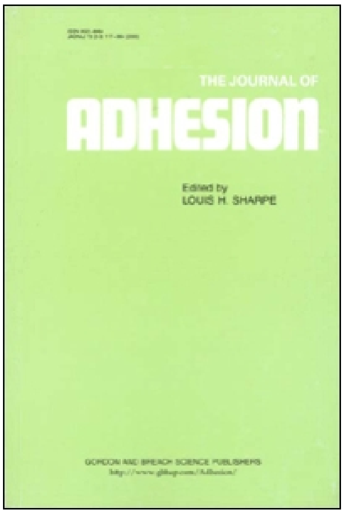

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The Interlaminar Bond Strength of Flexible Polymer-Metal Laminates

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An alternative test method to the traditional 180° "T" peel test has been developed for the measurement of interlaminar bonding in three-ply (polyester/aluminium/polypropylene) flexible-packaging, laminate materials. The new test is thought to involve mixed mode I (opening) and mode II (shearing) failure, and takes into account the yielding of the polypropylene layer during testing. The method allows more direct comparisons between materials to be made, and allows the measurement of strong bonds, where a peel test would result in yield or fracture of the substrate arms before debonding.

KEY WORDS Polymer-metal; polypropylene; laminate; peel; interlaminar bonding; adhesive bonds; interfacial strength; bond strength; bond energy.

1 INTRODUCTION

Flexible laminated packaging materials are becoming popular in many applications and especially in the food industry. Combinations of polyolefins, aluminium foil and polyester films have been heat-sealed into pouches, which have to withstand extreme conditions (such as those of retort pouches). It is thought that the strength of the adhesive bond between layers of a laminate may play a key role in determining the strength of the heat-seal region of a pouch formed by these laminates. To investigate the effect of the strength of the interlayer adhesive bond upon laminate performance, a suitable test method must be employed.

There are several standard test methods available for the measurement of adhesive bonds, each using a specific specimen geometry. Appropriate geometry and test conditions must be chosen to model the eventual service conditions. A review of the different standard test geometries has been given recently by Kinloch.[1]

For the flexible laminate packaging materials studied here the most appropriate standard test method is generally accepted to be a 180° "T" peel test.[2] However, there are limitations to this method. It is often difficult to prepare suitable test pieces. For high strength bonds the substrate arms yield and/or fracture in preference to failure of the adhesive bond. Also the measured "bond strength" can include components due to the yielding and bending of the substrates, as well as the breaking of the adhesive bond. It has been found[3,4] that the measured "bond-strength" in such a test arrangement is highly dependent on strain rate, thickness of substrates and the angle of the free tail ("peel angle").

An alternative test arrangement has been developed, which is thought to involve mixed mode I (opening) and mode II (shearing) failure. This models the failure mode in tests on heat-sealed polypropylene/aluminium/polyester laminates, where failures occurred along the polypropylene/aluminium interface. It is similar to the cracked lap shear (CLS) specimen developed by Johnson, Mangalgiri and Mall,[5,6] except that the CLS test relates only to rigid substrates.

The new test method has been employed to study a particular laminate system consisting of polyester/aluminium/polypropylene-copolymer layers, with a polyurethane/organic-solvent adhesive system between the layers. The method utilises the high elongation-to-failure of the polypropylene layer. Also, a component for the yielding of the polypropylene is subtracted from the measured "strength," and problems due to substrate bending are eliminated. The yielding behaviour of the substrate is determined by following a separate experiment.[7]

This new test method could also be used for laminates with other highly extensible polymer films (*e.g.* polyethylene) and for other adhesive systems (*e.g.* water-based, or hot-melt). The new test gives a measure of the adhesive bond energy, per original laminate area, between the polypropylene and aluminium layer.

The effects of test parameters—such as test-piece geometry, cross-head speed and test temperature—have been investigated in this study, to verify the assumptions made, and as a preliminary to standardisation of the test method. The final conditions chosen and the results of the test parameter investigation are presented in this paper. Finally, the test has been used to compare the behaviour of experimental laminates with that of commercially-produced materials.

2 THEORY—CALCULATION OF BOND ENERGY

2.1 Overall Bond Energy

The test-piece used is similar to a tensile film test,[7] but the laminate has a groove cut through the polyester/aluminium layer, Fig. 1. When the sample is loaded in tension the load-displacement curve shows a sharp peak, as the polyester/aluminium layer is completely fractured, followed by a plateau load as the polypropylene (PP) layer yields and the polypropylene/aluminium bond is broken. The interpretation of the load-displacement curve is given in Fig. 2.

It can be assumed that, at any interval during the test, an infinitesimally small section of PP deforms in the vicinity of the region undergoing debonding. Since the region of the film is relatively wide, there is a high width/thickness ratio, so that the PP is undergoing constrained plane strain deformation on this region. To determine the component of the overall load required for the deformation of the PP it is necessary to determine the properties of the non-laminated PP film. If a standard piece of unlaminated PP film is tested in tension, the load-displacement curve has

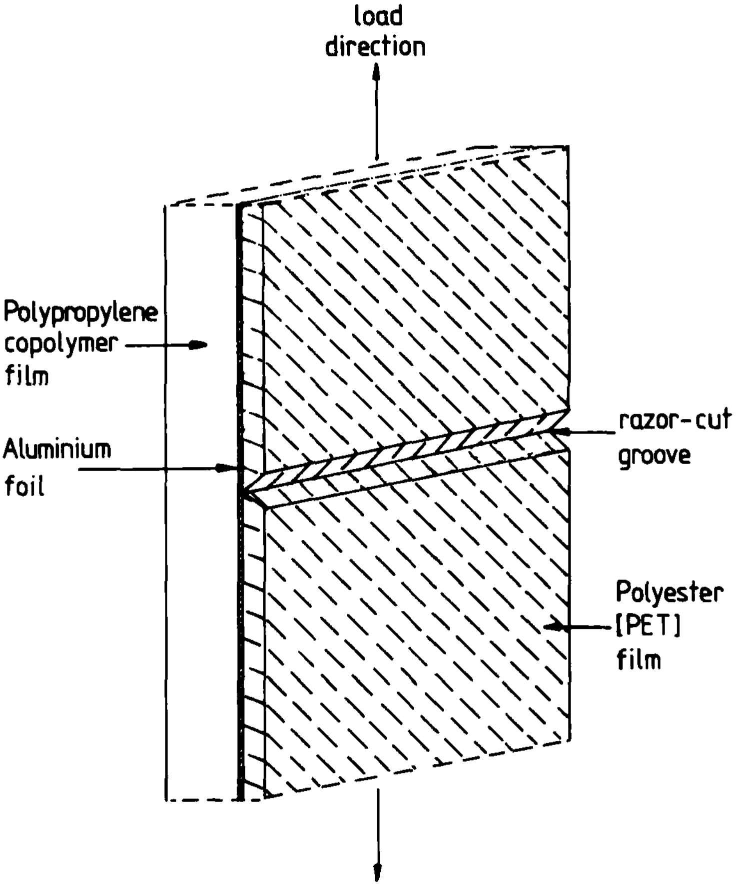


FIGURE 1 Test-piece showing groove through the polyester/aluminium layer of the laminate.

a peak load as the yielding is initiated, followed by a plateau load as the yielded area propagates. A typical load-displacement curve for PP is shown in Fig. 3.

If the gauge length of the film sample is altered, there is a change in the load-displacement curve. Fig. 4 shows the effect of gauge length on the peak and plateau loads for the PP film tests. The general trend is that as the gauge length decreases the peak and plateau loads increase and the width of the peak decreases. If the infinitesimally small section which undergoes plane strain deformation is considered as a sample of zero gauge length, the load required to propagate the yielding in a segment of zero gauge length can be determined by plotting the plateau loads of different gauge length PP film tests, and extrapolating back to zero, by using a computer to fit a polynomial to the experimental data.

Once the load for yield at zero gauge length of PP film (P_{opp}) has been determined, this can be used with the overall bond test plateau load (P_B) to calculate the bond energy. The area under the bond test load-displacement curve is a measure of the

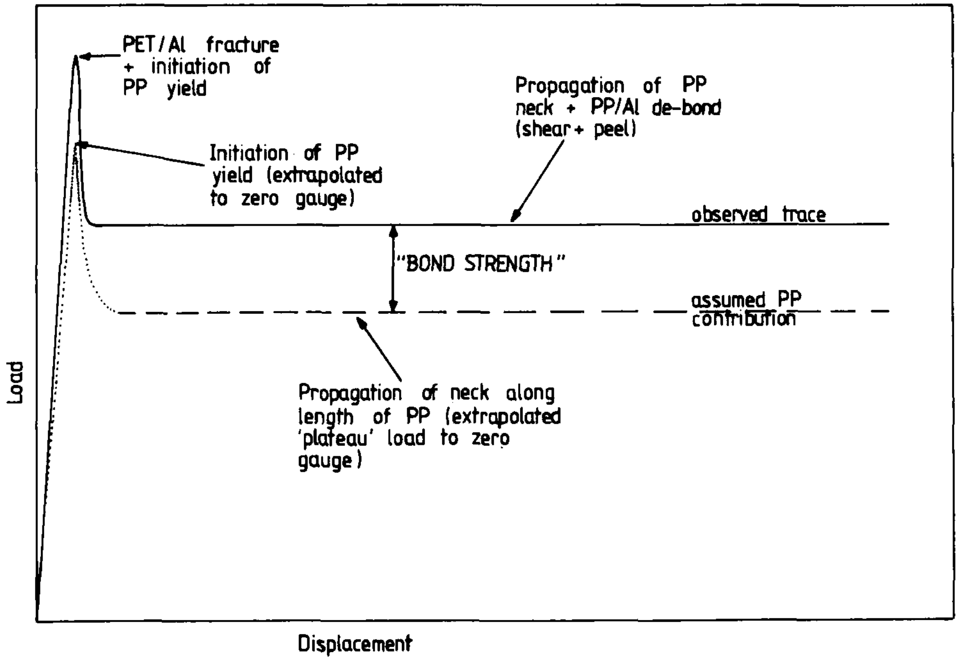


FIGURE 2 Interpretation of the bond test load-displacement curve.

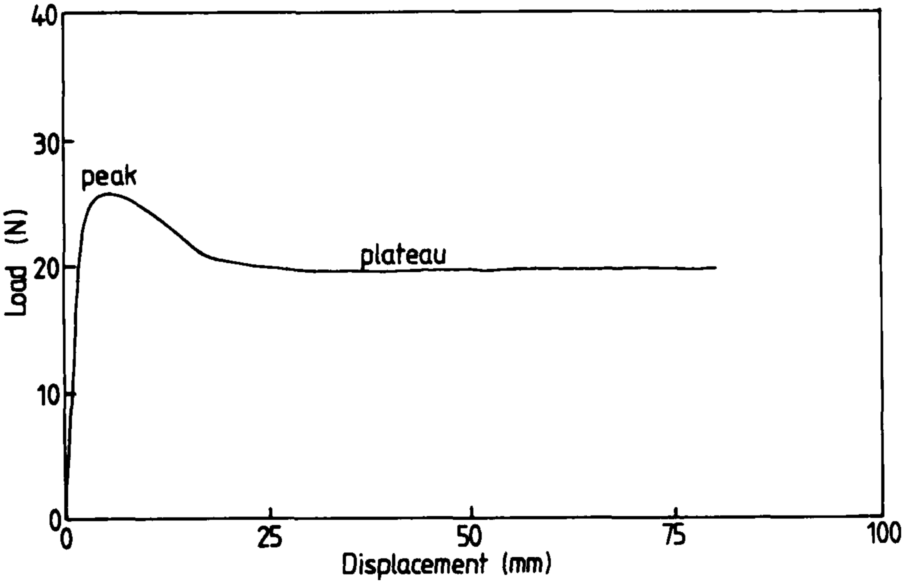


FIGURE 3 Typical polypropylene film load-displacement curve (for sample 25 mm wide, 50 mm gauge length, tested at room temperature and 50 mm/min)

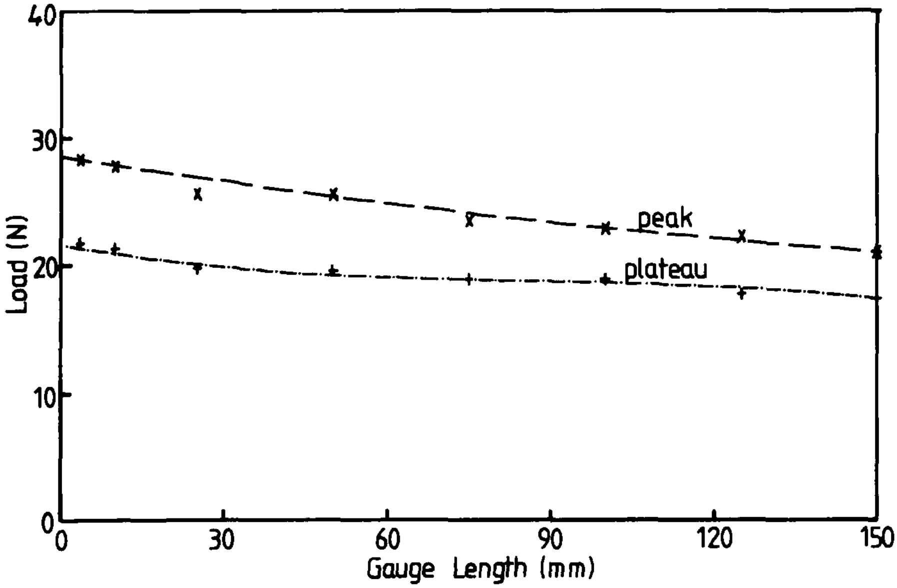


FIGURE 4 The effect of polypropylene film gauge length on peak and plateau loads. Samples 25 mm wide, tested at room temperature and 50 mm/min.

energy needed both to yield the polypropylene film and to separate an original laminate area, A, of the polypropylene/aluminium bond. The final displacement, X_f, can be taken as the difference between the end point on the load-displacement chart and the displacement at the initial peak, to correct for the initial break through of the polyester/aluminium and elastic deformation. The total energy absorbed, E_t, is then given by:

$$E_t = X_f \times P_B \tag{1}$$

The component for the yielding of the PP film, E_{pp}, is given by:

$$E_{pp} = X_f \times P_{opp} \tag{2}$$

The bond energy, E_B, is then given by:

$$E_B = \frac{E_t - E_{pp}}{A} \tag{3}$$

If

$$\Delta P = P_B - P_{opp} \tag{4}$$

then

$$E_B = \frac{X_f \cdot}{A} \Delta P \tag{5}$$

3 EXPERIMENTAL

3.1 Materials Used

Initial studies were carried out on a 3-ply polyolefin/aluminium/polyester laminate. The polyolefin film was a propylene-ethylene block copolymer (PP), which had been extruded without additives to produce a film approximately 70 μm thick. There was no surface pretreatment of the film.

The aluminium foil (Al) was approximately 9 μm thick, standard annealed foil. The polyester layer was a commercial 12 μm thick poly(ethylene terepthalate) (PET) film. The layers were laminated by a small-scale commercial laminator using a two-part, room-temperature curing, polyurethane adhesive. The laminate was passed through a drying oven at 70°C and then stored for 5 days at 25°C to complete the cure.

3.2 Test-Piece Geometry

The test-piece dimensions are given in Fig. 5 and each bond-energy test-piece was prepared in the following manner. A strip of laminate was cut a few millimetres wider than the final width required, and 50 mm longer than the required gauge length (to allow for gripping in the jaws). For consistency, the length of the strip was always parallel to the rolling direction of the laminate. A sharp razor blade was used, with light pressure, to score a groove through the polyester/aluminium layer, perpendicular to the length and approximately mid-way along the length. Care was taken not to damage the polypropylene layer. The test piece was then trimmed to the required width, with a sharp blade, to prevent any tearing from the edge during testing.

Samples of the polypropylene film only were also prepared. If the pre-lamination film was available this was used as received, otherwise the layers of the laminate were separated by dissolving the aluminium layer with acid (*e.g.* 5M HCl (aq)). Samples of various gauge lengths, (10 mm, 20 mm, 30 mm and 50 mm) and the appropriate width were prepared. Again, the total sample length was gauge length + 50 mm and the length of the sample was parallel to the rolling direction of the film. All test-pieces were mounted on to window cards, as shown in Fig. 5, using a latex adhesive, to prevent tearing at the jaws.

3.3 Tensile Testing

A displacement-controlled mechanical testing machine (Instron model 1122) was used. Initial work was carried out in normal laboratory conditions, but fluctuations in room temperature were found to have a significant effect on the results (see section 4.2.2) so subsequent work was carried out in a controlled-environment room (temperature 22.5 ± 0.5°C and relative humidity $50 \pm 5\%$), for the room temperature work, with the specimens being conditioned for 24 hours in the room before testing. An environmental chamber was used for the high temperature tests. The

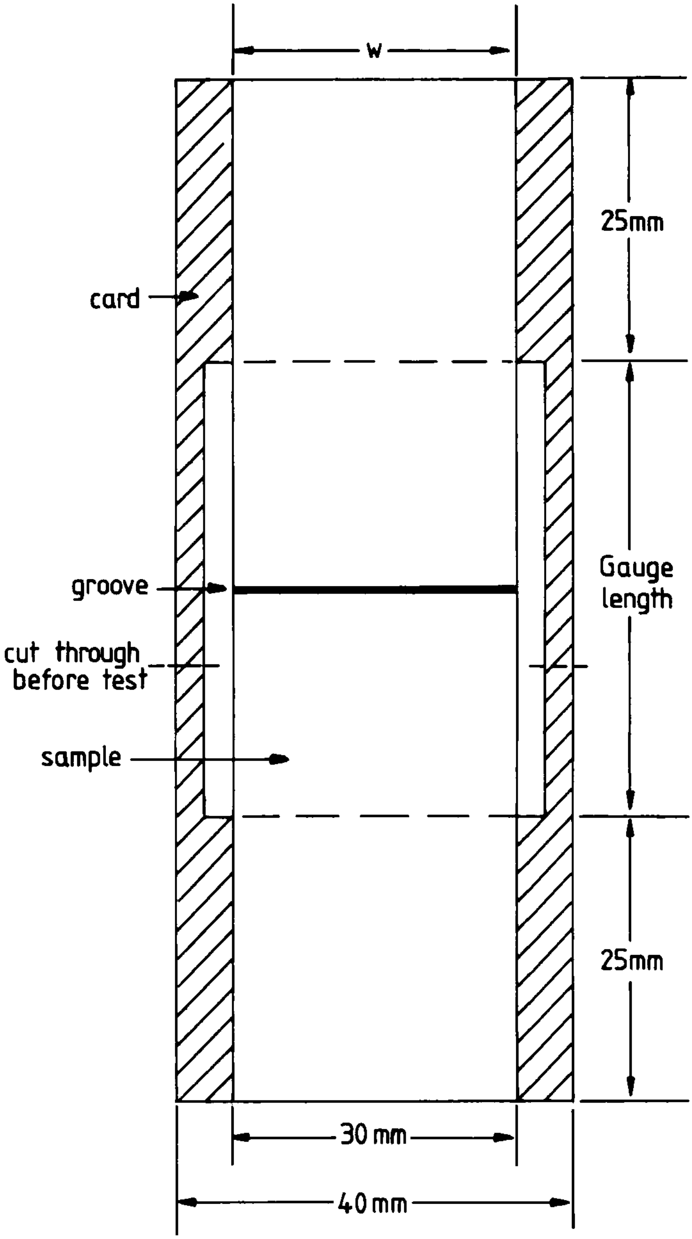


FIGURE 5 Geometry of test-piece showing method of mounting on window card.

standard cross-head speed used was 50 mm/min,[7] unless the effect of speed was being investigated.

Five test-pieces of each gauge length of polypropylene film were tested under the chosen test-speed and temperature conditions. The plateau loads (Fig. 3) were recorded for each test-piece and the mean and standard deviations calculated for each test-piece geometry. A number of bond strength test-pieces were then tested under the same conditions until at least 5 had failed in the "required manner," *i.e.* a rapid fracture of the aluminium/polyester layer, followed by yielding of the polypropylene layer, accompanied by debonding of the polypropylene/aluminium interface (and a curling of the polyester/aluminium layer away from the polypropylene). Tearing or fracture of the polypropylene layer was considered an invalid test, as was yielding of the "bonding laminate" or re-adhesion ("tacking") of the aluminium to the freed polypropylene adhesive. These failure modes invalidated the assumptions used to calculate the bond strength. The "bond-strength" tests were arbitrarily taken to a final displacement of 80 mm, however, as will be shown in section 4.1.4, the final displacement does not affect the measured bond-strength. The plateau load (see Fig. 2) for each "bond-strength" test-piece was recorded.

In order to evaluate the effect of final elongation on the measured bond strength, a series of tests were carried out, where each test-piece was tested to a different final elongation. For a more comprehensive study, intervals of 5 mm wide were marked on the PP film of the "bond-strength" samples with permanent ink, above and below the PET/Al groove. During the test, event marks were made on the load-displacement curve each time the edge of the debonded area reached one of the ink marks on the PP film (see Fig. 6). The bond energy for each interval (E'_B) was calculated by using the following equation:

$$E'_B = \frac{\Delta x}{\Delta A}(P_b - P_{opp}) \qquad (6)$$

where Δx is the difference in displacement measured from the chart, ΔA is the change in debonded (original laminate) area, calculated from a plot of chart displacement *versus* debonded length (see Fig. 6), and P_b is the main plateau load of the bond test in that interval.

3.4 Birefringence Examination

The deformed samples from the tensile tests on the films and from the "bond-strength" tests, were examined with cross polarised light. A polarising filter was placed on top of a Tungsten-filament light box, the samples were laid out flat on top of the filter, and were viewed with a 35 mm camera with a polarising filter over a macro lens. The birefringent characteristics of the materials provides information about the stress distribution and molecular orientation in the deformed polypropylene films.

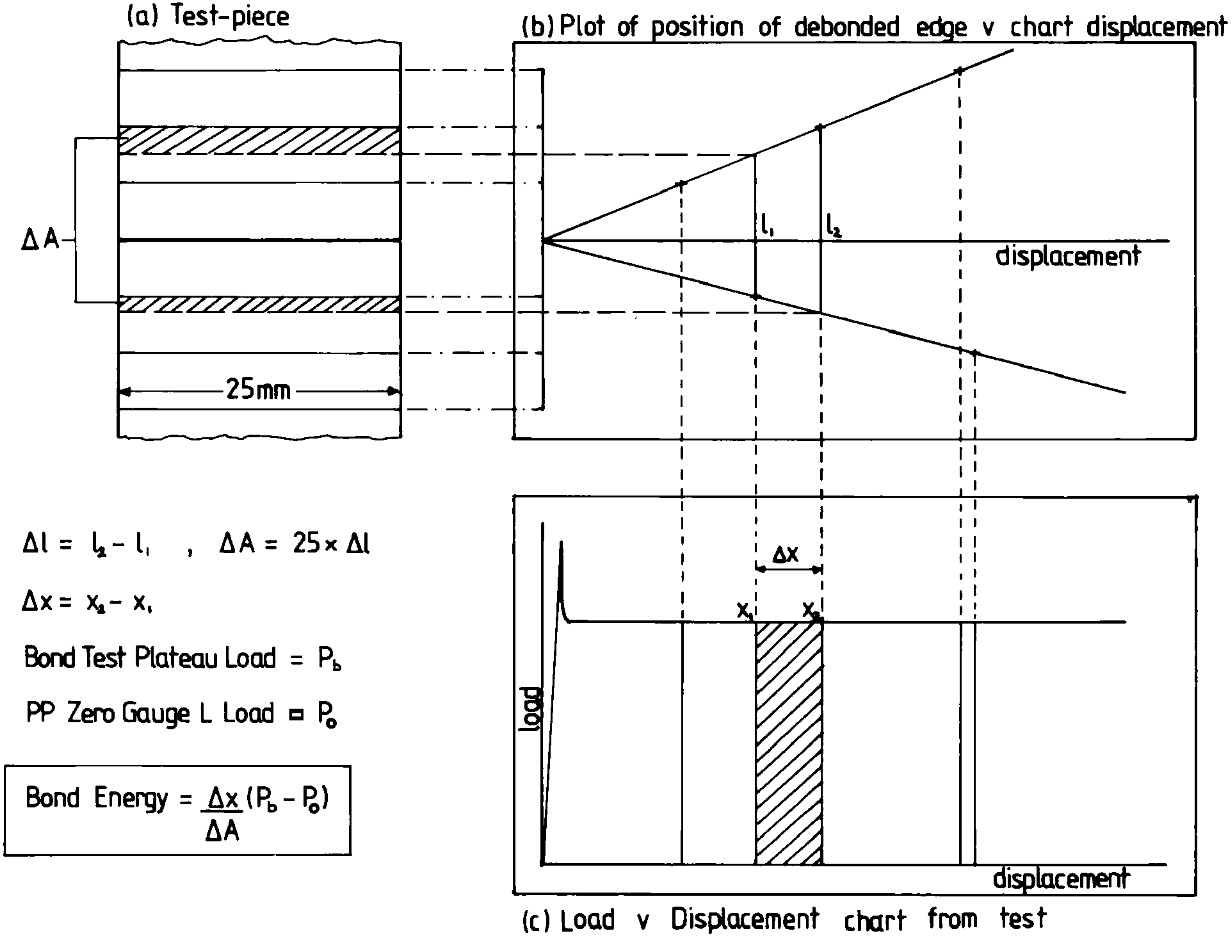


FIGURE 6 A representation of the method used to calculate the incremental bond energy (E'_B), to show the effect of final displacement.

4 RESULTS

4.1 Effect of Test-Piece Geometry

4.1.1 Effect of test-piece width Table I gives the bond energies calculated for different width samples, tested at room temperature at a cross-head speed of 50 mm/min. The results are also shown in Fig. 7. It can be concluded that in the range 15 to 30 mm there is no significant effect of the specimen width on the bond energy. Below 15 mm wide there is a significant drop in the bond energy. This is thought to be because the mode of failure changes, *i.e.* plane strain conditions no longer prevail, and edge effects become important.

TABLE I
The effect of test-piece width on measured bond energy. [Values are mean (standard deviation), all test-pieces 50 mm gauge length, tested at 50 mm/min temperature of 18–20°C]

Width (mm)	$\Delta P/w$ (N/mm)	A/X_f (mm)	Bond energy (J/m^2)
10	0.34(0.02)	7.53(1.0)	1154(151)
15	0.48(0.03)	7.35(0.7)	1657(212)
20	0.40(0.04)	6.56(0.2)	1511(150)
25	0.40(0.02)	6.86(0.1)	1584(107)
30	0.37(0.03)	6.40(0.1)	1426(128)

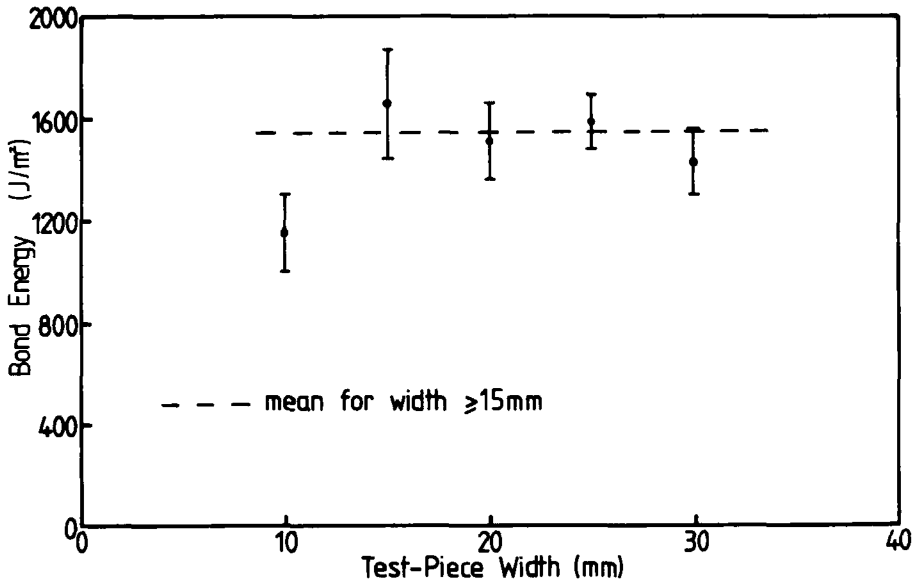


FIGURE 7 The effect of test-piece width on measured bond energy (E_B). All samples 50 mm gauge length, tested at room temperature and 50 mm/min.

4.1.2 Effect of test-piece gauge length Different gauge length "bond-strength" test-pieces were tested at room temperature and 50 mm/min. The same value of P_{opp} was used for each gauge length, since the assumption for the calculations is that a zero gauge length section of PP film deforms at any instant in time. The dependence of bond energies upon gauge length are given in Table II, and shown in Fig. 8. As expected, no significant dependence of bond energy upon test-piece gauge length can be seen.

4.1.3 Birefringence observations As the gauge length of the PP film sample decreases, the loading conditions approach that of plane strain. When a tensile load is applied under plane strain conditions, the sample width does not decrease significantly, causing preferential reduction in film thickness. Fig. 9 shows different

TABLE II
The effect of test-piece gauge length on measured bond energy. [Values are mean (standard deviation), all test-pieces were 25 mm wide and tested at a cross-head speed of 50 mm/min]

Gauge L (mm)	ΔP (N)	A/X_f (mm)	Bond energy (J/m^2)
20	9.0	6.37(0.3)	1409(162)
50	8.5	6.86(0.1)	1211(71)
75	9.1	6.62(0.1)	1368(69)
100	8.7	6.90(0.1)	1234(97)
150	8.9	6.60(0.1)	1347(144)

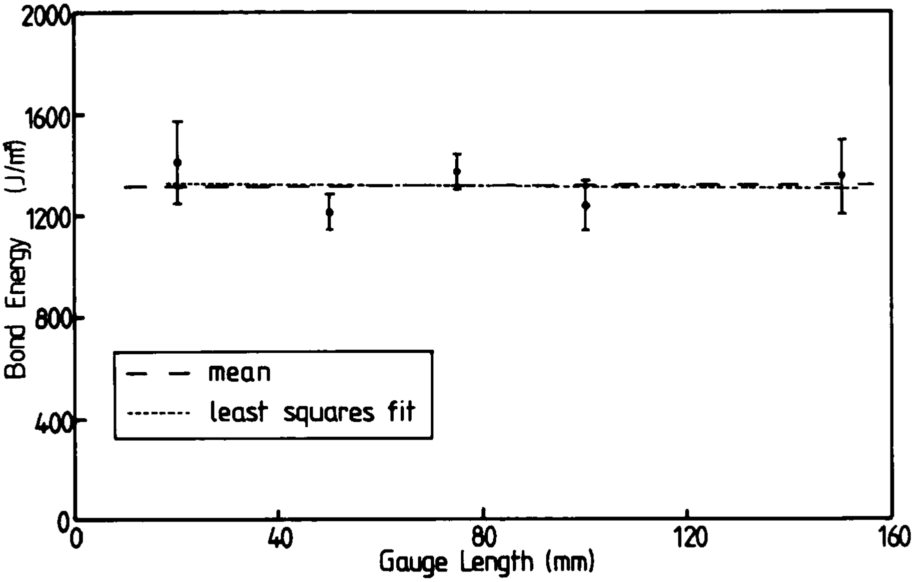


FIGURE 8 The effect of test-piece gauge length on E_B. All samples 25 mm wide, tested at room temperature and 50 mm/min.

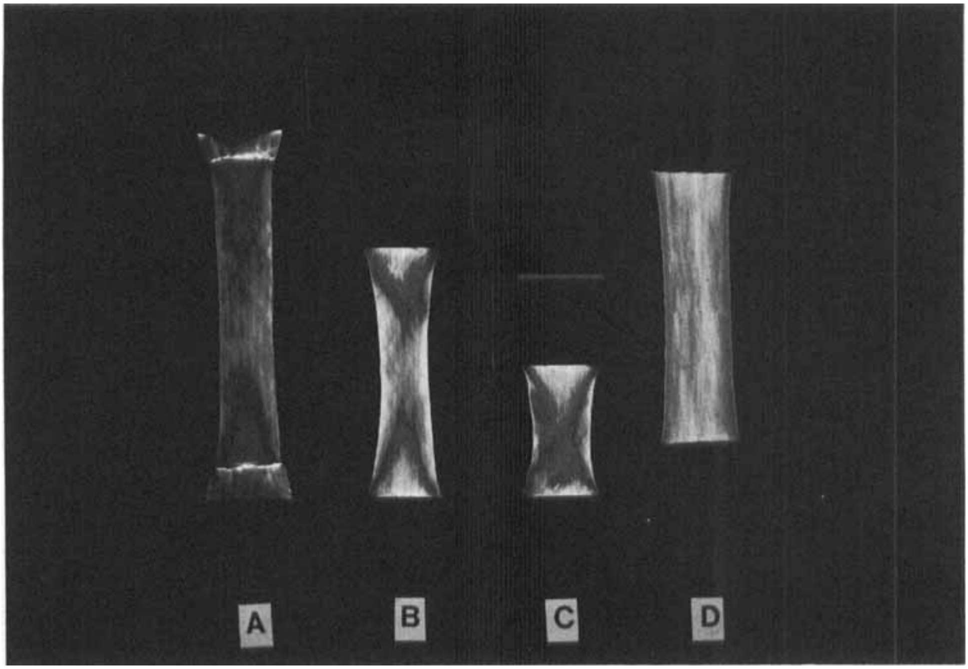


FIGURE 9 Samples of different gauge length polypropylene films tested at 50 mm/min at room temperature (A = 50 mm, B = 30 mm, C = 10 mm, D = bond test sample). Micrographs obtained using cross-polarised transmitted light.

gauge length, deformed PP films compared with a deformed bond-strength test-piece. The transition from plane stress to plane strain can be seen from the birefringent behaviour. Fig. 10 shows bond-strength samples which were tested to different final elongations. All the samples have the same appearance in the deformed film region, *i.e.* an orange colouration with blue edges (because of increased stress at the edge), together with a slight reduction in width. The lowest gauge length PP film sample has a very similar appearance to this.

4.1.4 Effect of final elongation Table III gives the results from the bond tests of specimens taken to different final elongations. These are shown in Fig. 11, with the mean line. It can be seen that there is a good correlation between the mean and the least squares fitted line, and it can be assumed that the final displacement has no effect on the measured bond energy. The results obtained by using the marked intervals are given for each test speed, as discussed in section 4.2.1, in Figs. 12 (a) to (d). The even scatter of the results about the mean line confirms the independence of bond energy on the final displacement.

4.2 Effect of Test Conditions

4.2.1 Effect of Test Speed The results for the different gauge lengths of polypropylene film, tested at four different speeds (20, 50, 100 and 200 mm/min), are given

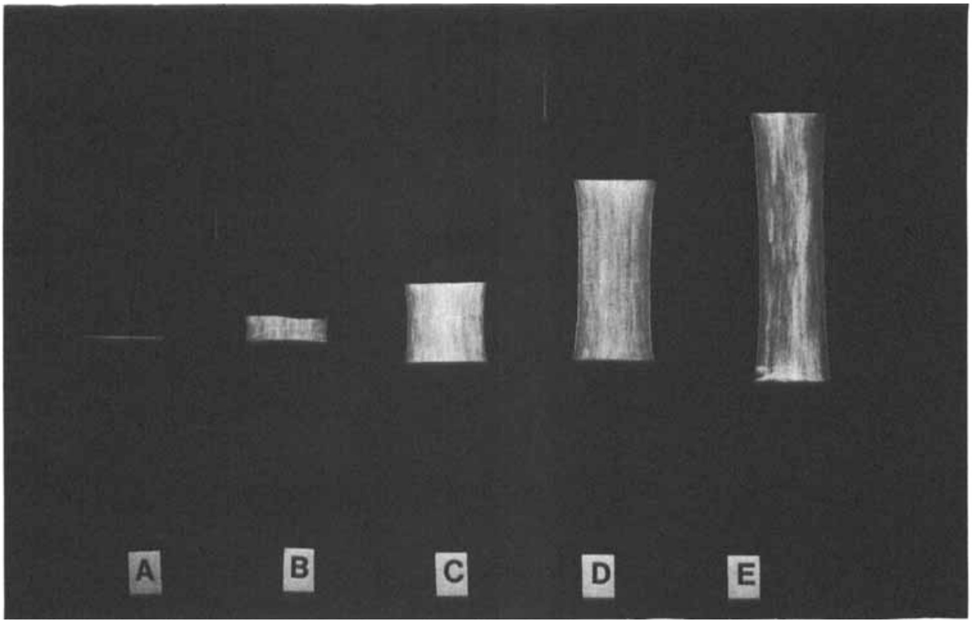


FIGURE 10 Bond test samples taken to different final elongations, tested at room temperature and 50 mm/min. Micrographs obtained using cross-polarised transmitted light.

TABLE III
The effect of final elongation (displacement) on measured bond energy, [All test-pieces were 50 mm gauge length, 25 mm wide, and tested at a room temperature of 23°C using a cross-head speed of 50 mm/min]

X_f (mm)	ΔP (N)	A/X_f (mm)	Bond energy (J/m^2)
44.7	7.3	6.34	1153
46.6	7.8	7.08	1105
62.0	7.2	6.74	1071
67.2	7.2	6.70	1078
77.3	7.1	6.60	1071
97.0	7.5	6.50	1157
97.8	6.6	6.78	978

in Table IV. A computer-calculated third order polynomial fit to the data was used to determine the extrapolated value of P_{opp}, also given in Table IV and Fig. 13. It can be seen that P_{opp} increases as the cross-head speed increases as might be expected for a viscoelastic polymer film.

The calculated values for P_{opp} were used with the experimentally determined values of P_B, given in Table V, to calculate the bond energy at different test speeds, also given in Table V. The mean and distribution (1 × SD) of the bond energy values are shown in Fig. 14. It can be seen from Table V that the value of P_B also increases with cross-head speed, and it can be concluded from these results that there is no

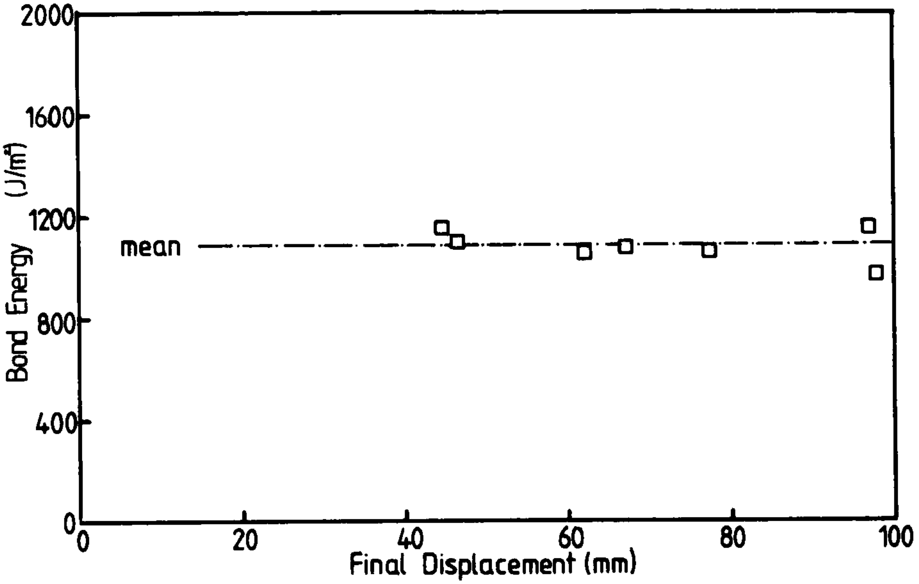
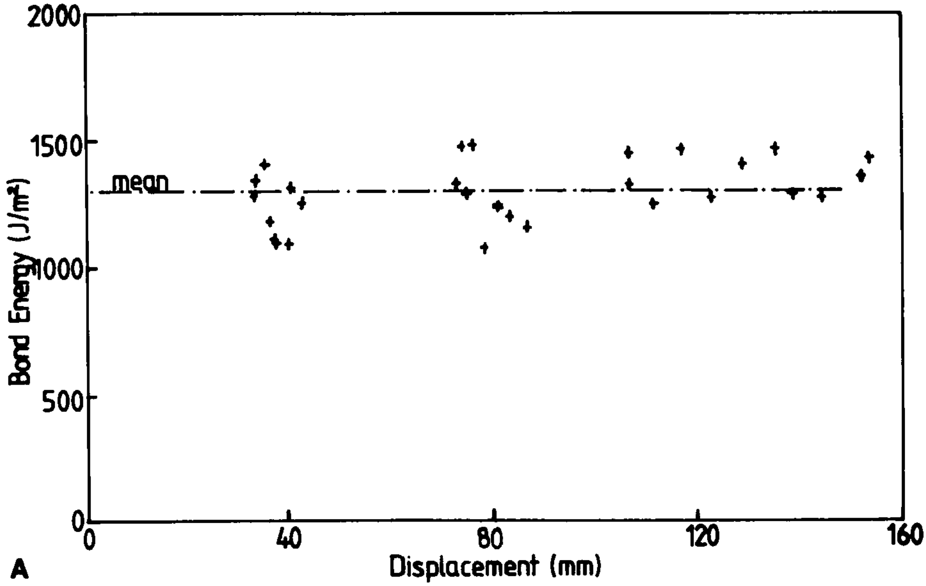
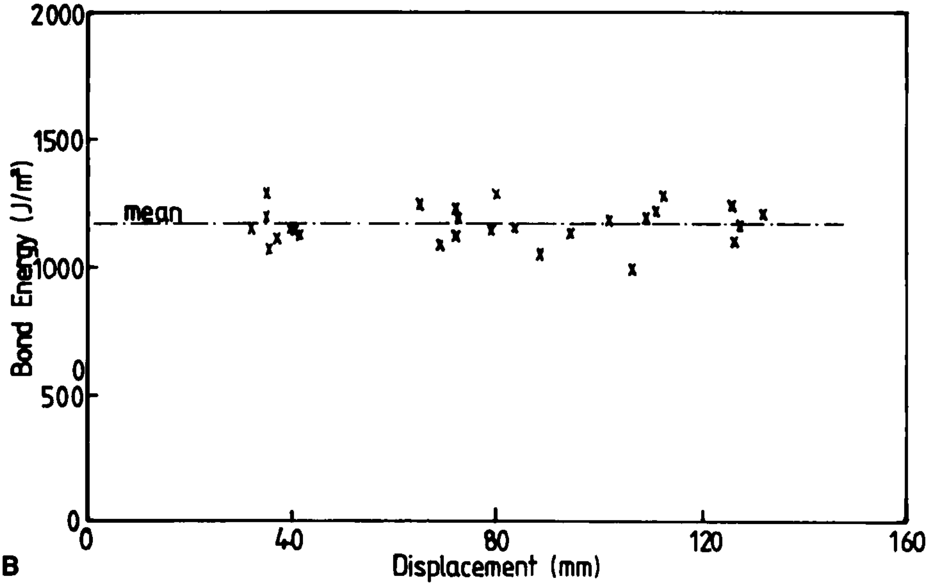


FIGURE 11 The effect of final displacement on E_B. All samples were 50 mm gauge length, 25 mm wide, tested at room temperature and 50 mm/min.



Caption for Fig. 12 on page 168.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1000
500
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mean
Bond Energy (J/m²)
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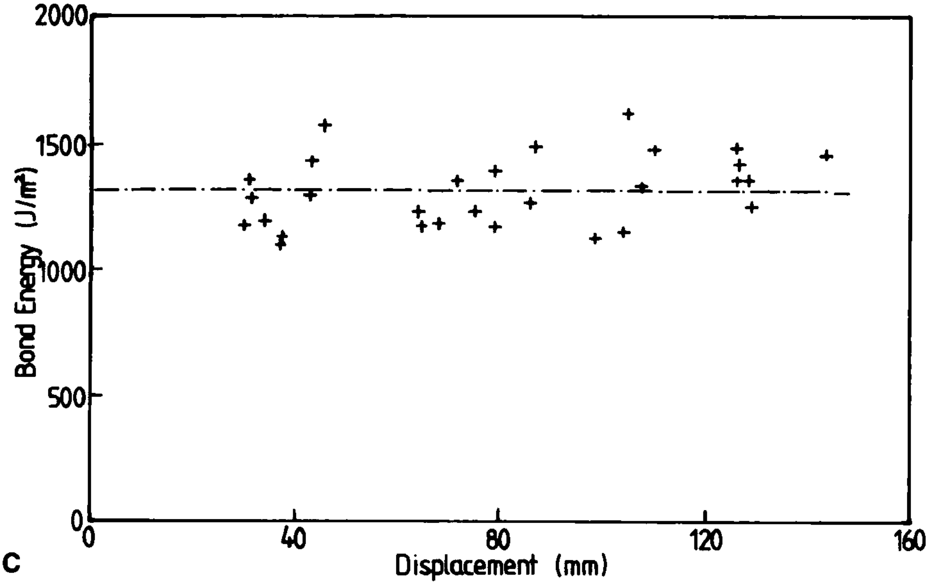
40
80
120
160
Displacement (mm)

B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Bond Energy (J/m²)
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Displacement (mm)

C

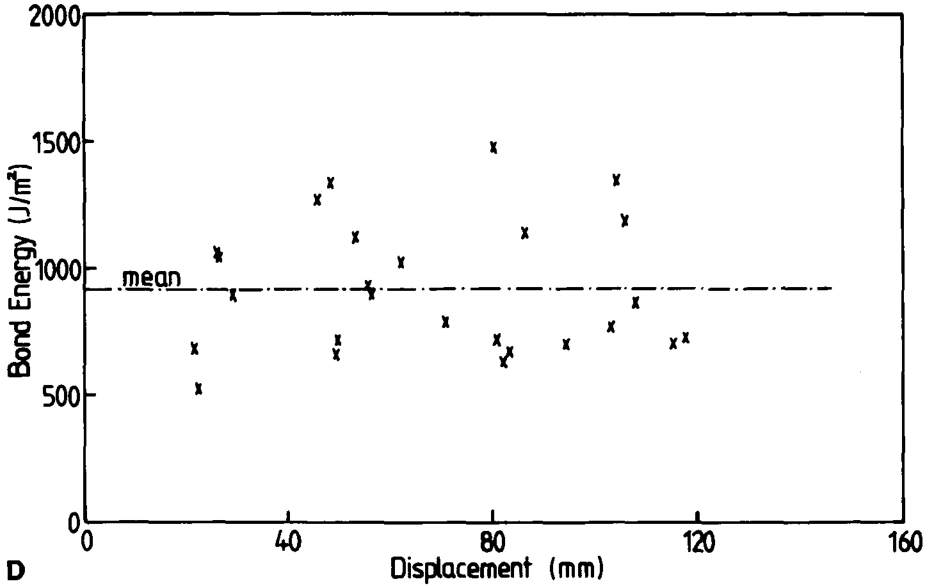

FIGURE 12 The scatter of results obtained by incremental calculation (eq 6) on tests at different speeds. (a) 20 mm/min (b) 50 mm/min (c) 100 mm/min (d) 200 mm/min. All samples were 50 mm gauge length, 25 mm wide and tested at 22.5 ± 0.5°C.

TABLE IV
The mean (standard deviation) plateau loads (in N) for different gauge length PP films, tested at different speeds, and at 22.5 ± 0.5°C

GL (mm)	Speed			
	20 mm/min	50 mm/min	100 mm/min	200 mm/min
50	15.6(0.2)	16.3(0.4)	18.4(0.4)	19.1(0.3)
30	16.4(0.5)	16.8(0.5)	18.2(0.4)	19.0(0.3)
20	16.9(0.6)	17.5(0.6)	18.5(0.1)	19.7(0.5)
10	17.6(0.7)	18.6(0.3)	19.3(0.3)	20.6(0.8)
Extrap. $O = P_{opp}$	18.4	20.0	20.4	22.0

significant variation in the calculated bond energy with test speed in the range 20–100 mm/min. However, there is a noticeable drop in the calculated bond energy, and an increase in the scatter of results (increase in SD) at the higher speed of 200 mm/min. This could be a result of adiabatic temperature rises during testing (see effect of temperature 4.2.2) or a change of mode of failure.

4.2.2 Effect of temperature The behaviour of the polypropylene film and the laminate PP/Al bond energy has been evaluated at different temperatures. The

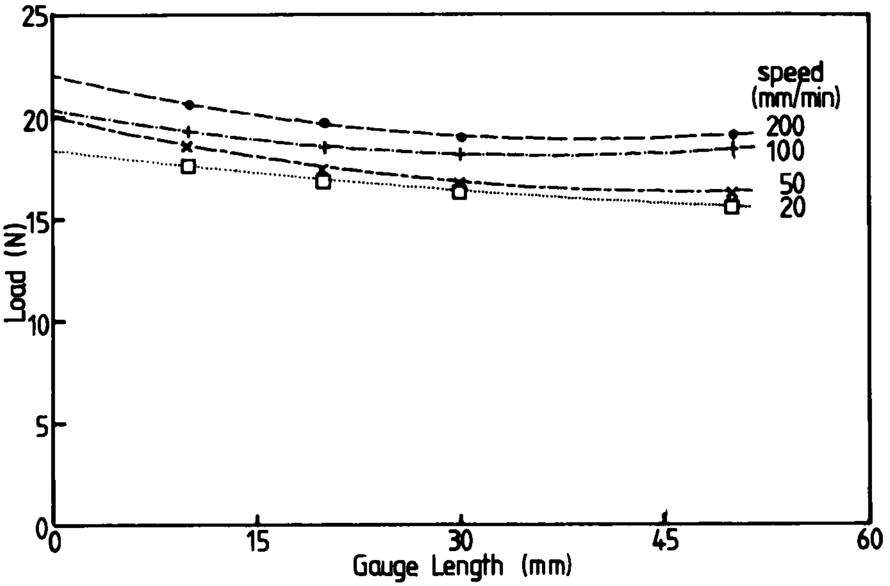

FIGURE 13 The effect of test speed on the behaviour of PP film and hence the value of P_{opp}. All samples were 25 mm wide and tested at 22.5 ± 0.5°C.

TABLE V

The effect of test speed on measured of bond energy [Values given as mean (standard deviation)]

Speed (mm/min)	Plateau load (N)	A/X_f (mm)	Bond energy (J/m^2)
20	26.7(0.5)	6.34	1354(71)
50	27.3(0.4)	6.30	1164(56)
100	28.6(0.6)	6.37	1298(90)
200	28.2(1.3)	6.71	919(178)

effect of temperature on the behaviour of the film is indicated in Table VI, and shown in Fig. 15. As expected, the yield strength of the film decreases with increasing temperature. The values of P_B decrease with increasing temperature, but not at the same rate as the decrease in P_{opp}. The effect of temperature on the bond energy is given in Table VII and Fig. 16.

For the particular laminate system studied, there was a significant decrease in the bond energy in the range 18 to 40°C, followed by a slight increase as the temperature increased above 40°C. This trend corresponds to an observed change in failure mode. Below 40°C the polyester/aluminium layer peeled away and the PP film yielded. Above 40°C the whole laminate contracted in a direction perpendicular to the applied load, leading to crinkling of the samples (and hence invalidating the assumptions). It is possible that there was further curing of the adhesive at elevated temperatures leading to the increase in bond energy.

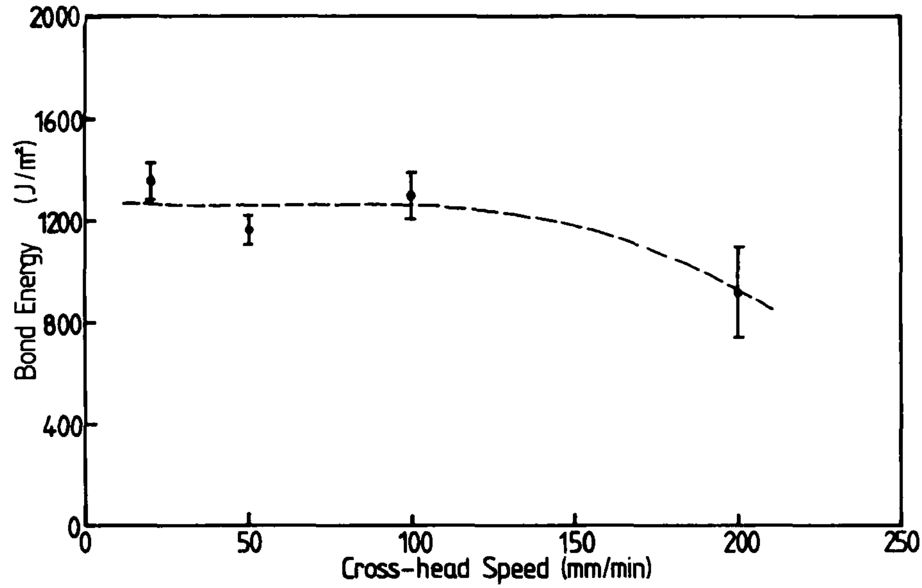

FIGURE 14 The effect of test speed on E_B. All samples were 25 mm wide, 50 mm gauge length, and tested at 22.5 ± 0.5°C. Error bar is ± 1 × SD and centre point (●) is the mean.

TABLE VI

The effect of temperature on the mean (standard deviation) plateau loads for 10 and 50 mm gauge lengths PP samples. [All test-pieces were 25 mm wide and tested at 50 mm/min]

Temperature (°C)	Plateau load (N) G.L. = 10 mm	Plateau load (N) G.L. = 50 mm
20	20.8(0.6)	19.5(0.2)
40	14.5(0.3)	12.0(0.4)
60	10.2(0.3)	8.5(0.3)
80	8.0(0.3)	7.0(0.1)

4.2.3 Comparison of materials The experimental laminate used throughout this investigation ("Experimental 1") was compared with a weakly bonded laminate ("Experimental 2") and two commercial laminates ("Commercial 1 and 2"). The weakly bonded laminate was produced in the laboratory by using a brush to coat a 2-ply PET/Al laminate with a water-based latex adhesive on the aluminium side, and then joining a random copolymer polypropylene film (70 μm thick). The commercial laminates were also 3-ply laminates of block copolymer PP film (50 μm), aluminium foil (9 μm) and PET (12 μm), and they were thought to be also bonded with polyurethane adhesives.

The bond energies for these laminates are given in Table VIII. It can be seen that the commercial laminates have higher bond strengths and a lower scatter in the data. This may be because the films used have surface pre-treatment, which increases

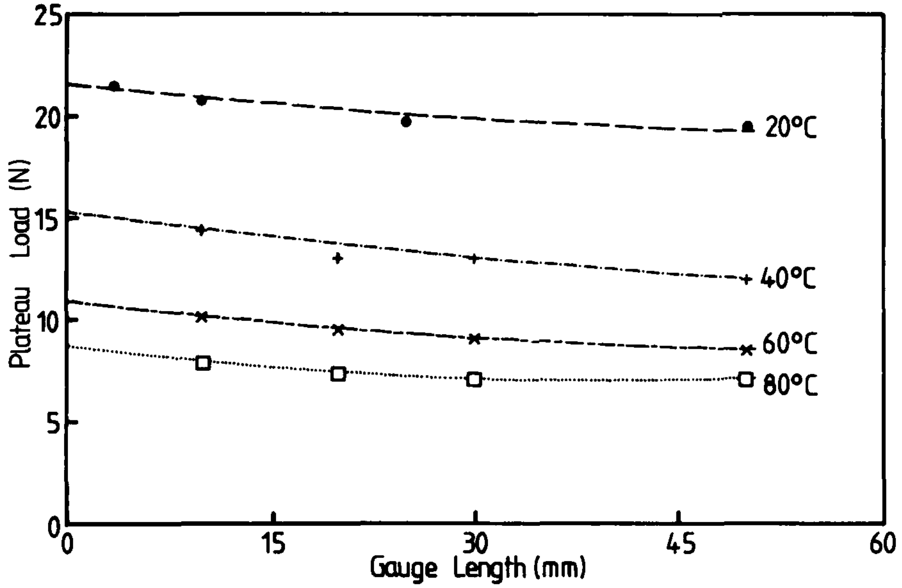

FIGURE 15 The effect of temperature on the plateau loads of the polypropylene film, and hence the value of P_{opp}.

TABLE VII
The effect of temperature on bond energy. [All test-pieces were 50 mm gauge length, 25 mm wide, and tested at cross-head speed of 50 mm/min]

Temperature (°C)	ΔP (N)	A/X_f (mm)	Bond energy (J/m^2)
18	10.1	6.34(0.2)	1584(107)
22.5	7.3	6.30(0.2)	1164(56)
40	3.6	6.75(0.1)	580(169)
60	5.0	6.99(0.1)	708(61)
80	6.2	6.66(0.1)	860(113)

interlaminar bond energy, and the scatter of data may be reduced because of the improved quality control of a large scale producer. The weakly-bonded laminate gives a lower bond energy and the crude method of production led to a greater scatter of results.

5 CONCLUSIONS

This new bond-strength test appears to be a useful method of comparing the bonding in polymer/aluminium laminates, particularly for high strength bonds, which are difficult to measure by other methods. The test has highlighted differences in bond energy between laminates, while taking into account differences in geometry and

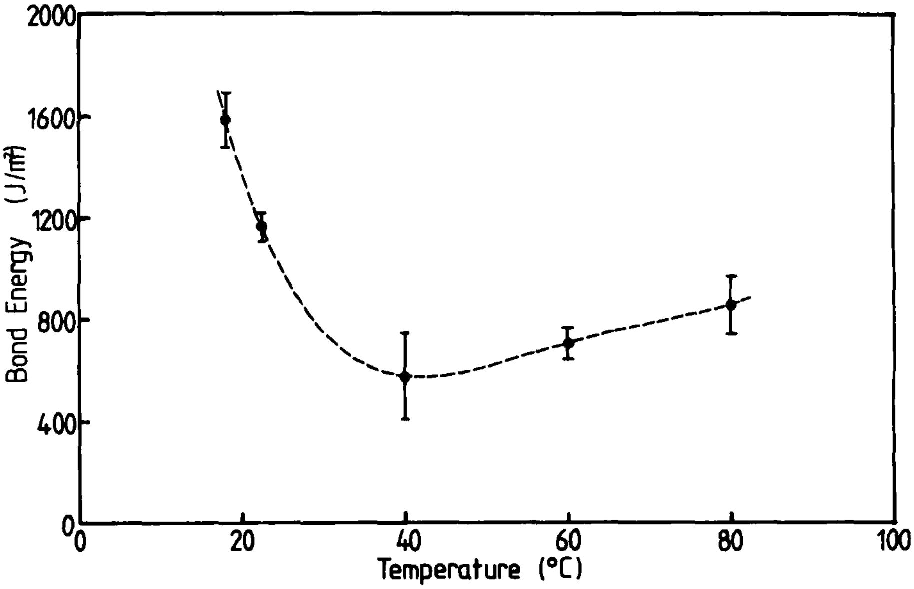

FIGURE 16 The effect of temperature on E_B. Error bar is ± 1 × SD and centre point (●) is the mean. All samples were 25 mm wide, 50 mm gauge length, and tested at 50 mm/min.

TABLE VIII
Comparison of bond energies of similar laminates. [All samples 50 mm gauge length, 25 mm wide tested at 50 mm/min and 22.5 ± 0.5°C]

Laminate	Mean plateau load (N)	P_{opp} (N)	Bond energy (J/m²)
Experimental 1	27.3	20.0	1164(56)
Experimental 2	23.4	15.9	895(127)
Commercial 1	33.7	22.8	1399(29)
Commercial 2	54.1	43.3	1415(80)

film thickness, etc., which can affect peel-test measurements. The investigation has shown that there is no significant effect on the measured bond energy when varying the sample width between 15 and 30 mm, or the sample gauge length between 20 and 150 mm. The test speed has no significant effect in the range 20 to 100 mm/min, as long as the separated films are tested at the same speed as the bonded samples. However, the temperature has been found to have a significant effect on the bond energy, so the temperature must be carefully controlled if comparisons are to be made between different laminates. This preliminary study could lead to the development of a standard test method, and may be useful both as a quality control method for the laminating industry and as a method of evaluating the fundamental adhesion characteristics between layers in laminates.

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